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XANES Analysis of Phosphorus Transformations in Soils Undergoing Redox Changes

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Introduction: Phosphorus discharges from agricultural lands to water bodies are a significant cause of water quality deterioration in many watersheds. The solubility of phosphorus in soils and sediments is affected by redox conditions through their impact on Fe-oxide minerals. The objective of the work undertaken at X-19A is to determine changes in the solid-phase P speciation in soils subjected to reducing conditions.

Methods and Materials: Soil samples were collected from agricultural fields in Quebec (Canada) and North Carolina (USA). In the laboratory, the clayey and silty soils from Quebec were subjected to three alternating reduction / oxidation cycles over 100 days. P K-XANES spectra were collected on the whole samples after each redox phase. Particle-size fractionation using sonification was also carried out on the samples to separate the silt and clay fractions which were submitted to XANES analysis. At NC State University, incubation experiments were performed on the silt and clay fraction of a sample of sandy surface soil from the eastern coastal plain of NC, which was subjected to reducing conditions in a continuous-stirred, controlled-atmosphere reactor. Sub samples of soil suspension were collected for XANES analysis at different time periods during reduction to follow changes in solid-phase P speciation over time.

Results: Both Quebec soils had similar levels of total P (51 mmol/kg) but differed in their pedogenesis. In the Providence soil, dissolved phosphate and iron concentrations increased during reduction phases. In contrast, the Dolbec B soil did not release phosphate during reduction. It was hypothesized that its high level in oxalate extractable Al, a redox inactive phase, might regulate the phosphate level in solution. XANES results showed distinctly different features for each soil. XANES spectra for the silt and clay fraction from Providence samples were characterized by an intense shoulder on the high-energy side of a strong white-line peak, while such a shoulder was not present in the Dolbec B samples. In both cases, changes in the intensity of the white line peak were observed following reduction and oxidation, indicating that the redox treatments induced transformation of the solid-phase P speciation. However, changes in the XANES spectra from both soils tended to diminish with successive redox cycles, suggesting that P transformations were less significant with each successive redox treatment, perhaps because of transformation of P onto non-active redox phases. In the surface soil sample from the eastern coastal plain of North Carolina, XANES spectra were similar for the sample before and after exposure to reducing conditions for up to 9 days (compared with a 30-day reduction period for the soil from Quebec), with a minor, but detectable decrease in the white-line peak after reduction. The dissolved phosphate in the reactor increased about 4-fold during this time period.

Conclusions: Sustained reduction or oxidation significantly affected the speciation of solid-phase phosphorus in two soil samples from Quebec, as indicated by changes in the intensity of the white line peaks of phosphorus K-XANES spectra. Sub samples of soil from NC collected at more frequent time intervals during reduction in a redox reactor showed very little change in XANES spectra up to 9 days.

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